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# **DESIGN AND EVALUATION OF IMPROVED BARRIER FABRICS FOR PROTECTION AGAINST TOXIC AEROSOLS AND BIOLOGICAL AGENTS**

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## Table of Contents

List of Figures .....	v
List of Tables .....	vii
Preface .....	ix
1.0 INTRODUCTION .....	1
2.0 MATERIALS AND METHODS	
2.1 Materials Studied .....	1
2.2 Microscopical Observations .....	1
2.3 Determination of Pore Size Distribution .....	2
2.4 Measurement of Aerosol Penetration .....	4
3.0 RESULTS AND DISCUSSION	
3.1 Photomicrographs .....	7
3.2 Pore Size Measurements .....	7
3.3 Aerosol Penetration .....	14
4.0 CONCLUSIONS .....	19
5.0 FUTURE WORK .....	19
6.0 REFERENCES .....	20
APPENDIX .....	21
Fabric Samples Examined in This Report	22

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## List of Figures

<u>Figure</u>	<u>Page</u>
1. Apparatus for Measuring Pore Size Distribution by Liquid Extrusion .....	3
2. Apparatus for Measuring Maximum Fabric Pore Size by Critical Pressure (bubble point) Method .....	5
3. Apparatus for Measuring Aerosol Penetration through Fabrics .....	6
4. Transmitted Light Photomicrograph of Tyvek Sample .....	8
5. Transmitted Light Photomicrograph of Polyester Sample .....	8
6. Fluorescent Microscopical Photomicrograph of Back of Nyco Fabric .....	9
7. Design of Nyco Twill Fabric .....	9
8. Pore Size Histograms of Fabrics SD1-SD5 Measured by Liquid Extrusion .....	10
9. Pores in Nyco 4.5 Fabric Observed with the Image Analysis System .....	12
10. Pores in Nyco 7 Fabric Observed with the Image Analysis System .....	13
11. Effect of Fabric Areal Density and Structure on Penetration of 2.21 $\mu$ m Diameter Latex Spheres .....	15
12. Penetration of Latex Spheres through Fabrics SD1-SD5 as a Function of Particle Diameter .....	16
13. Effect of Challenge Time on Particle Penetration of 2.21 $\mu$ m Diameter Latex Spheres through Fabrics SD1-SD5 .....	18

## List of Tables

<u>Table</u>	<u>Page</u>
1. Fabric Description and Particle Penetration .....	2
2. Fabric Mean Pore Size and Pore Size Ranges Measured by Different Methods .....	11
3. Penetration of Polystyrene Sphere Aerosols through Fabrics .....	17

## **PREFACE**

This report was prepared by the College of Textiles, North Carolina State University under U.S. Army Natick Research, Development and Engineering Center (Natick) Contract DAAK60-91-K-0010 - Phase 1.. The objective of this contract was to evaluate existing and experimental fabrics to determine their suitability for protection against aerosols. Evaluation methods include using aerosols of fluorescent polystyrene latex spheres to determine the mechanisms of particle capture together with several techniques for measuring the pore size distributions of the fabrics. The investigation reported was conducted during the period October 1991 to October 1992. Drs. Solomon P. Hersh and Paul A. Tucker were the Program Managers at NCSU and Ms. Marie Jean-Pierre and Dr. Donald Rivin were the Natick Project Officers.



**DESIGN AND EVALUATION OF IMPROVED  
BARRIER FABRICS FOR PROTECTION  
AGAINST TOXIC AEROSOLS AND BIOLOGICAL AGENTS**

## **1.0 INTRODUCTION**

The ultimate objective of this research is to develop semipermeable barrier fabrics which provide better protection for chemical protective clothing applications. In order to understand the relationship between the aerosol particle penetration and the structure of barrier fabrics, the research activities focused firstly on measuring the transmission of aerosols through test fabrics, determining the penetration mechanisms, and evaluating the pore size distributions in the fabrics. This report describes the work and results of testing and evaluation conducted during the past year.

## **2.0 MATERIALS AND METHODS**

### **2.1 Materials Studied**

Nine fabrics were selected for the study: spunbonded polyethylene nonwoven (Tyvek™), polyester woven with antistatic grid (PET woven), U.S. Army 4.5 oz/yd<sup>2</sup> and 7oz/yd<sup>2</sup> nylon-cotton fabrics (Nyc 4.5 and Nyc 7, respectively), and a set of five battle dress uniform fabrics (SD1, SD2, SD3, SD4, and SD5), which were supplied by the U.S. Army Natick RD & E Center. Samples of these fabrics are presented in the Appendix. Descriptions and characteristics of these fabrics are summarized in Table 1.

### **2.2 Microscopical Observations**

The structural characteristics of the barrier fabrics were determined by observing them using light and/or fluorescent microscopy. Samples of the test fabrics were mounted on a glass slide and examined with three types of microscopes: (a) a low resolution stereomicroscope using both reflected and/or transmitted light, (b) a compound transmitted light microscope, and (c) a compound back reflected fluorescent microscope. Photomicrographs were taken of each fabric. An image analysis system was used to characterize the pore structures of some of the fabrics.

**Table 1. Fabric Description and Particle Penetration**

Fabric	Fiber Content	Construction	Areal Density (oz-yd <sup>2</sup> )	Fabric Count Warp × Filling	Particle*	
					Penetration (%) Mean	S.D.
Tyvek	polyethylene	nonwoven (point bonded)	1.37	—	0.2	0.2
PET woven	polyester	woven-plain	3.39	134 × 89	45.3	2.7
Nyco 4.5	nylon/cotton	woven-twill	4.65	94 × 51	62.6	0.4
Nyco 7	nylon/cotton	woven-twill	7.30	85 × 55	38.6	2.3
SD1	cotton	woven-plain	6.18	104 × 54	32.6	1.3
SD2	polyester/cotton	woven-plain	6.76	108 × 58	29.0	3.4
SD3	nylon/cotton	woven-plain	5.97	106 × 52	37.2	2.0
SD4	cotton/Kevlar/ nylon	woven-twill	5.66	106 × 92	47.9	2.2
SD5	nylon/cotton	woven-twill	7.78	96 × 56	38.6	4.1

\* Using aerosol of 2.21  $\mu\text{m}$  diameter polystyrene spheres at a flow rate of 0.1 ft<sup>3</sup>/min and a face velocity of 1.8 cm/s, as described in the Results and Discussion section.

### 2.3 Determination of Pore Size Distribution

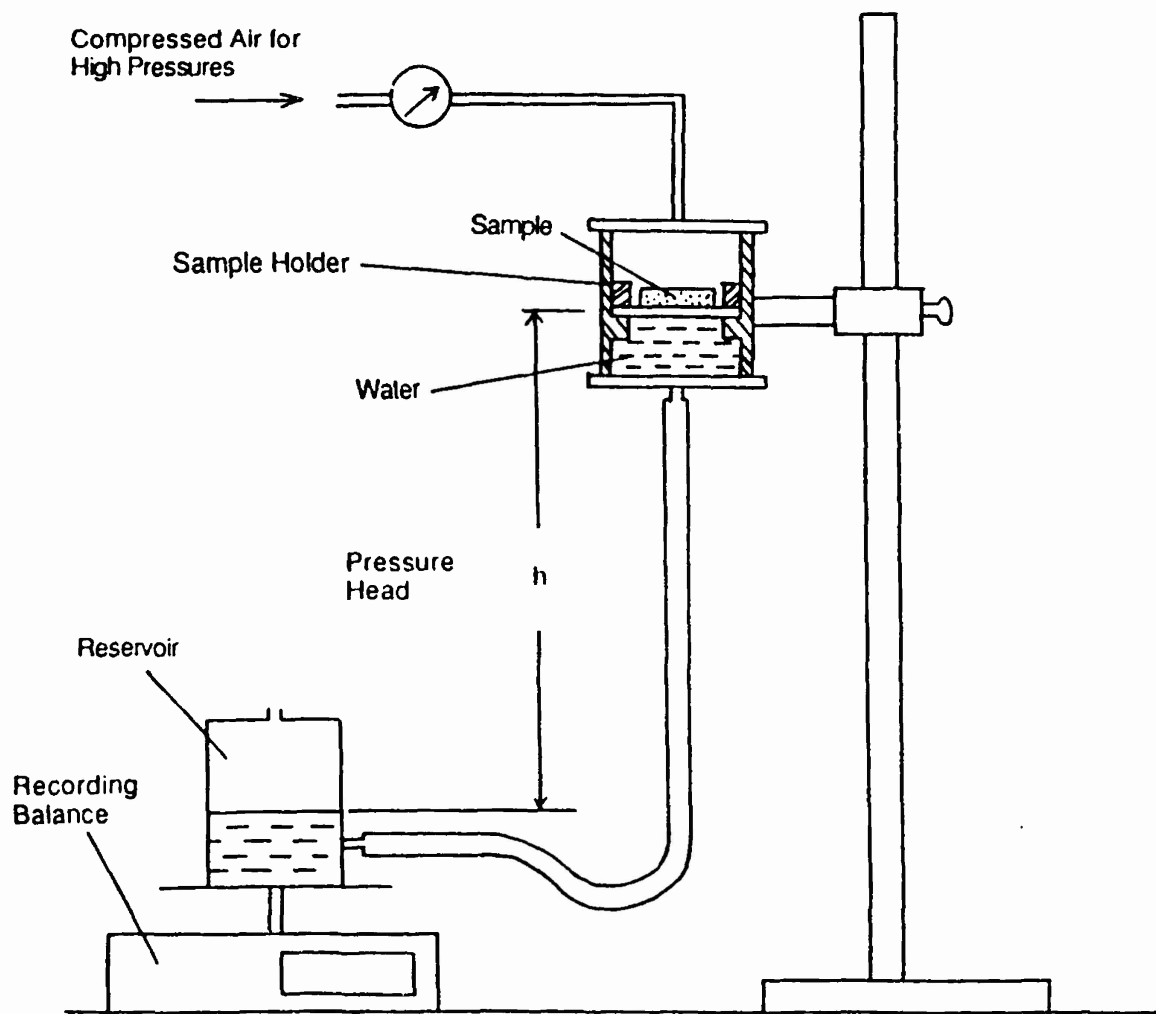
The distributions of pore sizes in the test fabrics were measured using the following three methods:

#### (a) Liquid extrusion

In the liquid extrusion method, the pore sizes are measured by first saturating the fabric with water and then measuring the pressure required to force the water out of the fabric pores. The pressure drop  $\Delta p$  required to force a liquid out of a pore of radius  $R$  is given by the LaPlace equation [1]:

$$R = 2\gamma \cos \theta / \Delta p$$

where  $\gamma$  is the surface tension between the air-liquid interface and  $\theta$  is the receding contact angle. A diagram of the experimental instrumentation is given in Figure 1. The fabric is pre-wetted in the liquid to ensure that all fabric pores are filled, and then the fabric sample is placed in a sample holder. A column of liquid is then maintained in contact with the sample through a sequence of flexible and rigid tubings connected to a reservoir placed on a top-loading recording balance. When the pressure gradient across the sample is increased by raising the sample holder, liquid is forced out of those pores that exceed a size  $R$  as defined by the LaPlace equation. The pores in the test fabrics are assumed to have circular cross sections. If the pressure required to express the liquid out of the smallest pores exceeds the pressure head "h" available, compressed air can be applied to reach



**Figure 1.** Apparatus for Measuring Pore Size Distribution by Liquid Extrusion.

higher pressures. The weight of liquid drained is then recorded each time the pressure gradient is increased. The volume fraction of pores having radius  $R$  is then obtained by dividing the weight of drained liquid at a specific height by the total liquid drained.

(b) Critical breakthrough pressure

The maximum pore size of the fabrics was determined by using a critical breakthrough pressure method [2]. This method is again based on the LaPlace equation. Here, the diameter of the largest pore passing from one surface to the other is determined with the apparatus illustrated in Figure 2. The fabric samples initially are thoroughly wetted in a liquid of known surface tension  $\gamma$ . Then, the sample is clamped into a sample holder connected to a pressure source. To initiate the test, sufficient liquid is added to just cover the sample, and the air pressure  $P$  through the sample is slowly increased until air bubbles are observed to first pass through the sample. The diameter of the maximum pore,  $D_{\max}$ , is then given by [2]

$$D_{\max} = 4\gamma \times 10^6 / \rho P g$$

which simplifies to

$$D_{\max} = 408 \gamma / P$$

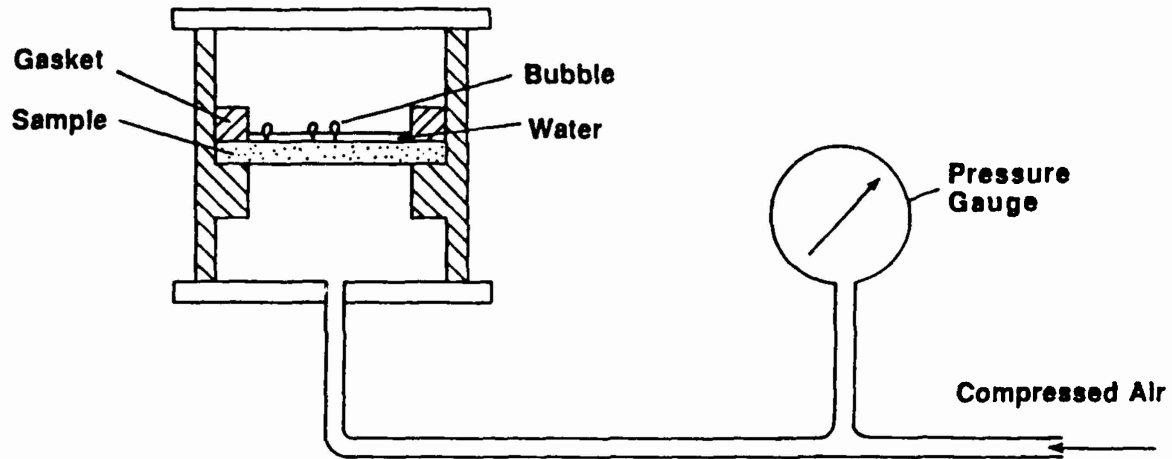
where  $D$  is in  $\mu\text{m}$ ,  $\gamma$  is in  $\text{mN/m}$ ,  $P$  is in  $\text{mm head of water}$ ,  $\rho$  is the density of water in  $\text{g/cm}^3$ , and  $g$  is the acceleration due to gravity. In the measurements reported here, the immersion liquid was water.

(c) Image analysis

The pore size distribution was also measured directly with an image analysis system. The fabric to be measured was placed on the stage of a microscope and illuminated with a perpendicular beam of transmitted light, and the areas of the pores in the test fabric were then determined from the observed images. The image was transmitted directly from the microscope via a video camera to the image analysis system. The number and size of the individual pores were then automatically counted and recorded by measuring the areas of light transmitted through the fabric.

## 2.4 Measurement of Aerosol Penetration

The penetration characteristics of test fabrics were assessed using polystyrene latex spheres of known diameter. Both fluorescent and nonfluorescent spheres were used, ranging in diameter from  $0.6 \mu\text{m}$  to  $4.5 \mu\text{m}$ . The instrumentation used is illustrated in Figure 3. A flux of polystyrene latex spheres was generated with a Climet Model CI-295 Aerosol Generator. The aerosol flux then was drawn through the test fabric clamped in the holder assembly at a flow rate of  $0.1 \text{ ft}^3/\text{min}$ , which yielded surface velocities of  $3.6 \text{ ft/min}$  ( $1.8 \text{ cm/s}$ ). The number of latex spheres penetrating the fabric were counted with a Met One Inc. Model A2300 laser particle counter. The pressure drop across the fabric was measured with a Magnehelic™ differential pressure gage. Percent particle penetration ( $P$ ) and filtration efficiency



**Figure 2.** Apparatus for Measuring Maximum Fabric Pore Size by Critical Pressure (bubble point) Method.

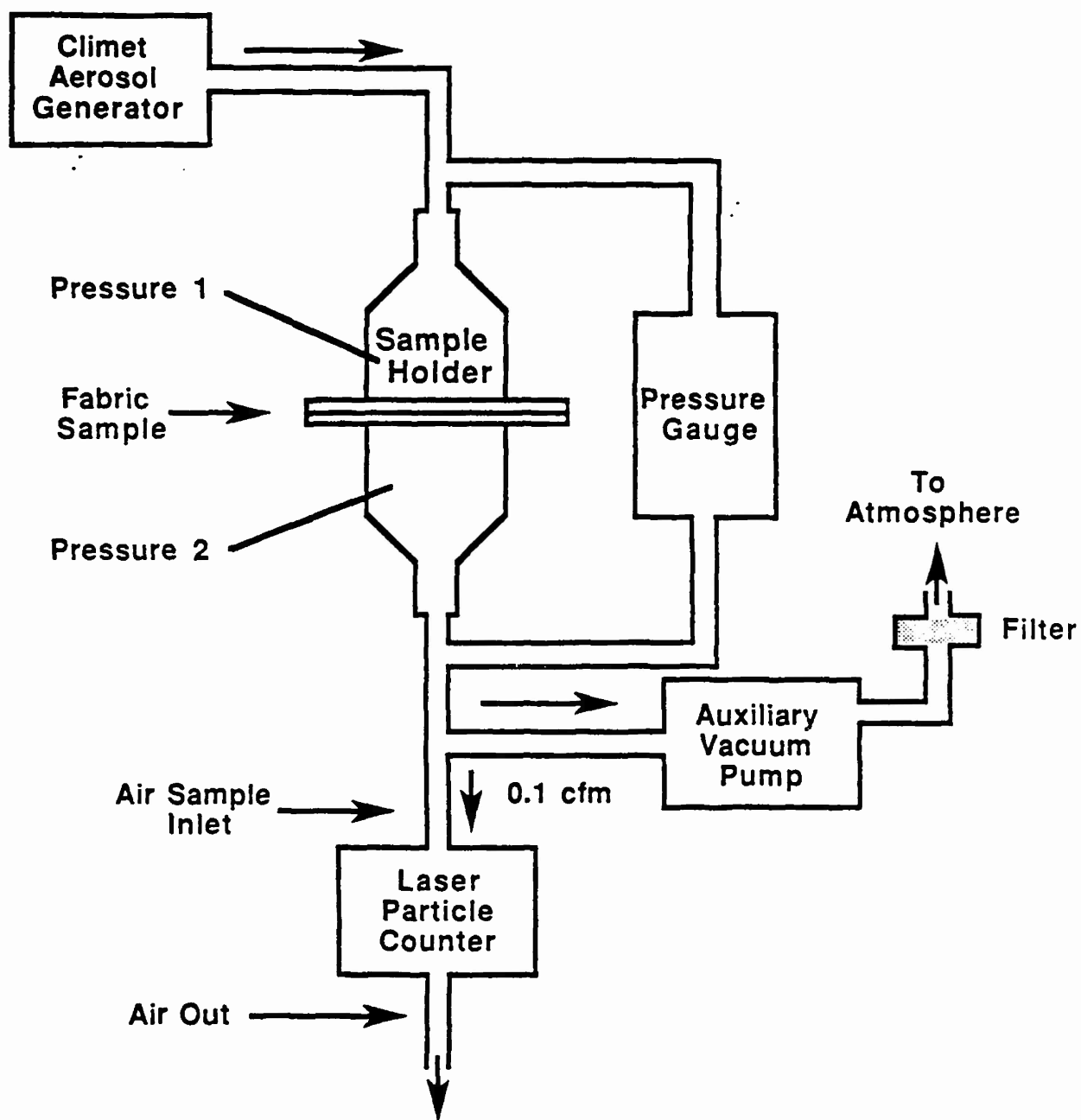


Figure 3. Apparatus for Measuring Aerosol Penetration through Fabrics.

(E) were determined by comparing the particle count with and without the test fabric in the holder using the following relationships [3]:

$$P = [(\text{Particle count with fabric}) / (\text{Particle count without fabric})] \times 100\%, \text{ and} \\ E = 100 (1 - P)$$

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Photomicrographs

Photomicrographs of the Tyvek and woven polyester fabric using transmitted light (quartz-halogen source) are shown in Figures 4 and 5, respectively. The white circular spots which appear in Figure 4 are the point-bonded areas in the Tyvek. Individual fibers cannot be easily distinguished in these spots, probably as a consequence of the melt bonding which likely occurs in these areas. The fibers in the surrounding areas, however, can be clearly seen. Because of the low magnification of this photomicrograph and the small pore size in the fabric, it is difficult to resolve visible pores in the photomicrograph. For the woven polyester fabric with antistatic grid, a very symmetrical plain weave pattern is seen in Figure 5, and many pores located at the interlacing of the yarns can be observed.

A photomicrograph of the Nyco 7 fabric is shown in Figure 6. For this picture, 0.60  $\mu\text{m}$  diameter polystyrene latex particles which emit yellow fluorescent light at 509 nm were applied to the face of the fabric. The back side of the fabric was then observed with a fluorescent microscope. In the photomicrographs of the treated fabric, the bright spots show the locations where fluorescent particles penetrated the fabric. These fluorescent spots form diagonal rows or strings which appear only along parallel oblique lines. This observation is a consequence of the twill structure of the Nyco 7 fabric, a 2:1 twill weave shown in Figure 7. The interlacing of the warp and weft yarns form pores at the crossover points. All positions where fluorescent particles permeated the fabric, as shown in Figure 7, are located at the pores formed at the interlacing points of the yarns. If the pore size is larger than the diameter of the fluorescent particles, some spheres will permeate through the fabric.

#### 3.2 Pore Size Measurements

The mean pore sizes and pore size ranges of the nine fabrics described above are presented in Table 2. The pore size distribution of the set of five woven battle dress uniform fabrics (SD1 through SD5) measured with the liquid extrusion method are shown in Figure 8. Except for fabric SD2, these fabrics have pores ranging from 60 to about 150  $\mu\text{m}$  in diameter with mean pore sizes around 90–110  $\mu\text{m}$ . The pore sizes of SD2 are smaller than those of the other fabrics. The mean pore size of this fabric is about 83  $\mu\text{m}$  with pores ranging from 40 to 90  $\mu\text{m}$ . Generally speaking, however, no great difference was observed in the mean pore size and pore size range of these fabrics.

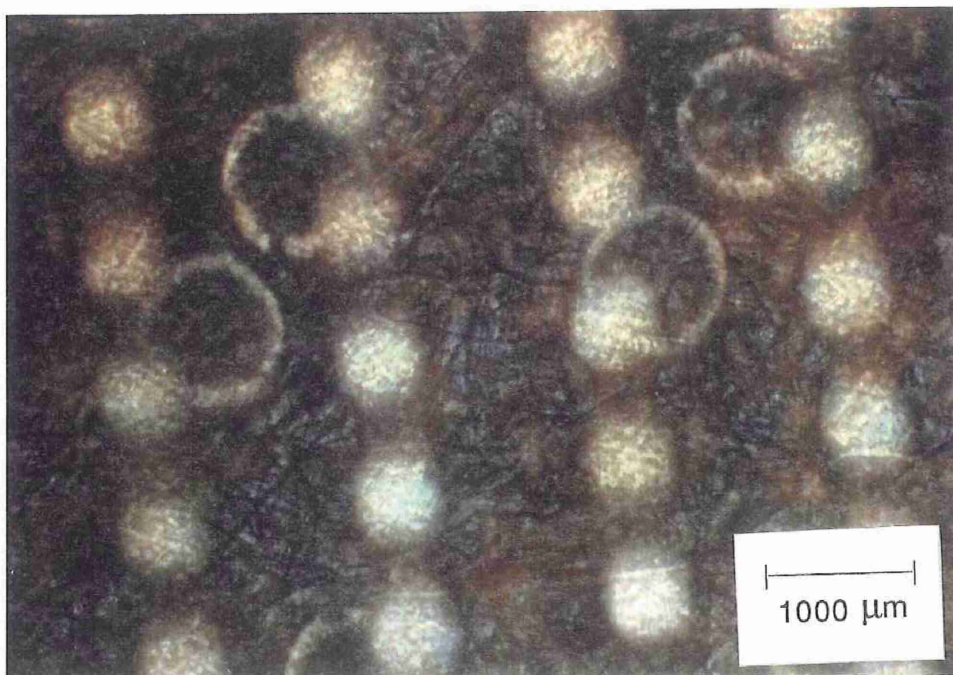


Figure 4. Transmitted Light Photomicrograph of Tyvek Sample.

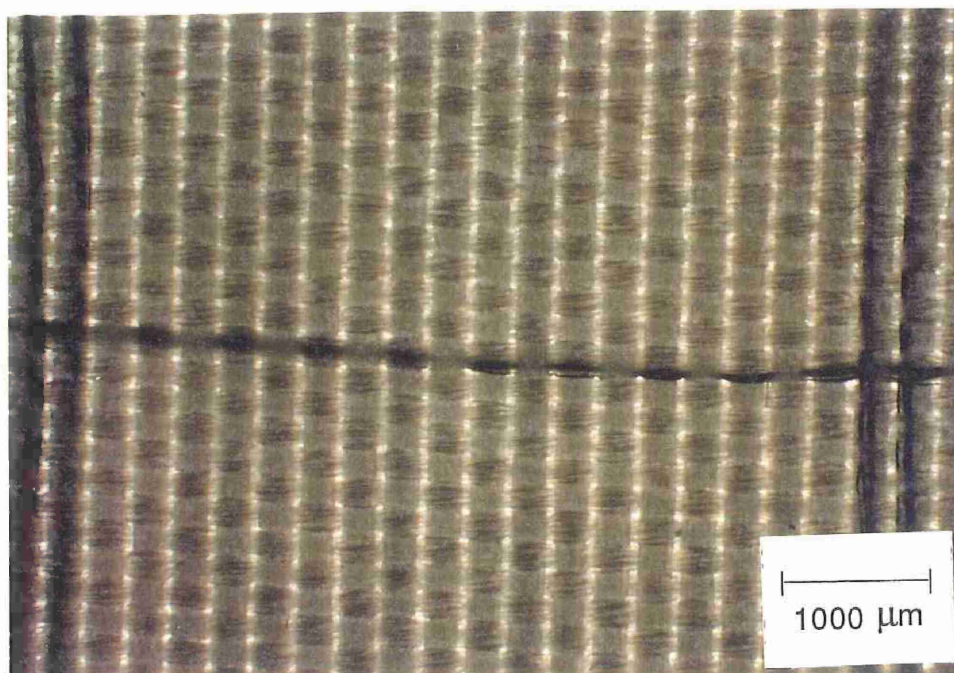
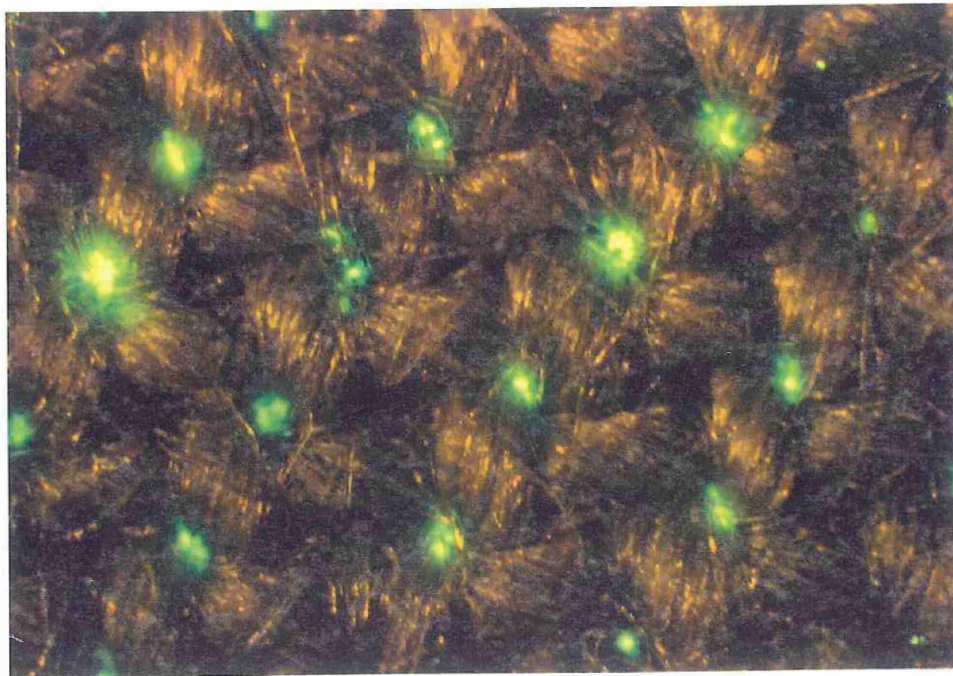
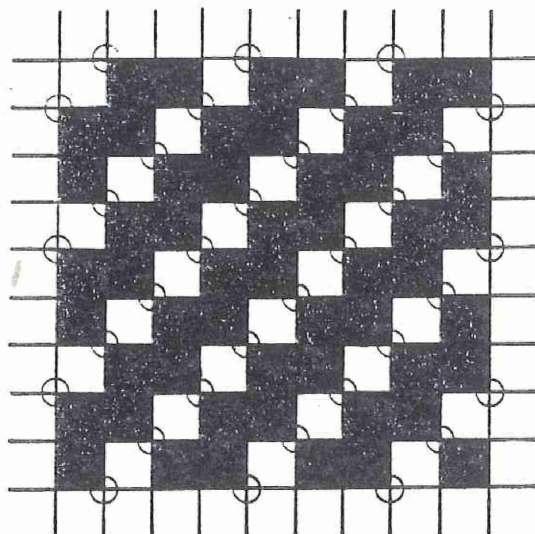


Figure 5. Transmitted Light Photomicrograph of Polyester Sample.

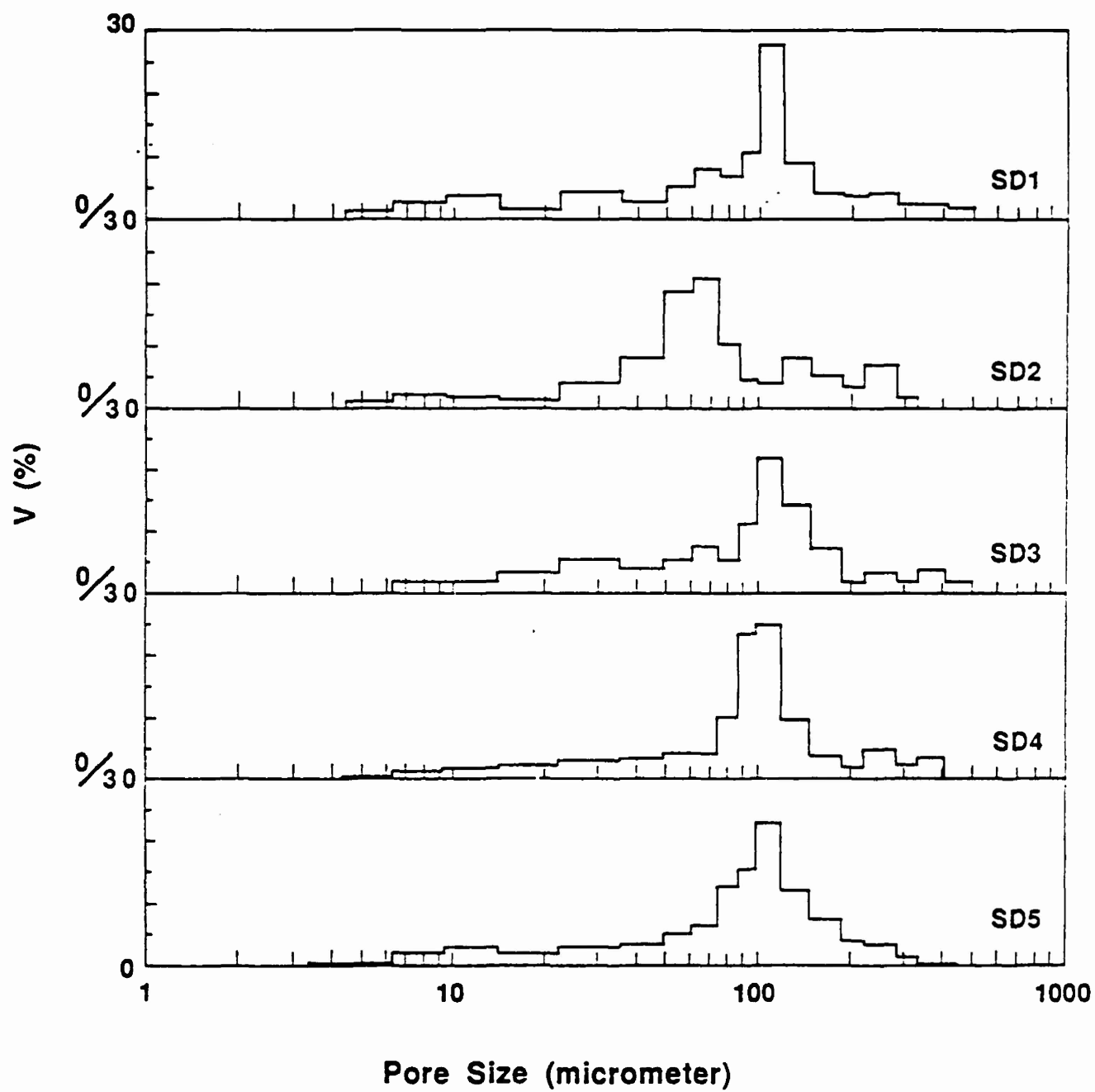




**Figure 6.** Fluorescent microscopical photomicrograph of back of Nyco fabric (warp yarns are in vertical direction). Bright spots are clusters of fluorescent particles that have penetrated the fabric.



**Figure 7.** Design of Nyco twill fabric. Small circles represent the location of fluorescent particles penetrating fabric as shown in Figure 6.



**Figure 8.** Pore Size Histograms of Fabrics SD1-SD5 Measured by Liquid Extrusion.

In order to corroborate these results, the pore size of fabrics was also determined by image analysis and critical breakthrough pressure methods. Typical image analysis results were obtained on the Nyco 4.5 and Nyco 7 fabrics and are shown in Figures 9 and 10 which have the same magnification. Although the actual shapes of the pores generally resemble collapsed ovals rather than circles, pore sizes are reported as equivalent pore diameter, or size, assuming the pore to be circles with the same area. As shown in Table 2, the mean pore size of the Nyco 4.5 fabric measured by image analysis was 104  $\mu\text{m}$ , with most of the pores ranging in diameter from 50 to 200  $\mu\text{m}$ . For the Nyco 7 fabric, the mean pore size is lower, 61  $\mu\text{m}$ , with a range from 10 to 100  $\mu\text{m}$ . Thus the latter fabric appears tighter and would suggest that the Nyco 7 fabric should have a higher filtration efficiency than the Nyco 4.5. This expectation was confirmed by the penetration data reported in Table 1. The penetration of fabric Nyco 4.5 (62.6%) is indeed greater than that of Nyco 7 (38.6%).

The pore sizes of test fabrics measured using the three different techniques are compared as shown in Table 2. The three sets of results obtained by liquid extrusion, critical pressure, and image analysis methods are reasonably consistent with each other, although the pore sizes measured by image analysis are lower than those measured with the other two. It is reasonable to expect lower values to be measured by image analysis because this technique depends on the amount of light passing through the fabric, some of which would be blocked by the tortuosity of the pores and their obliqueness to the fabric face. The other two methods should not be affected by these factors.

**Table 2. Fabric Mean Pore Size and Pore Size Ranges Measured by Different Methods\***

Fabrics	Mean pore size ( $\mu\text{m}$ )			Pore range ( $\mu\text{m}$ )		
	(LE)	(CP)**	(IM)	(LE)	(CP)**	(IM)
Tyvek	36	—	25	10-80	37	10-50
PET woven	74	—	25	40-150	62	10-50
Nyco 4.5	171	—	104	100-400	218	50-200
Nyco 7	97	—	61	60-150	135	10-100
SD1	102	—	86	60-150	111	16-230
SD2	83	—	49	40-90	87	16-137
SD3	107	—	47	90-150	126	14-143
SD4	103	—	41	70-120	97	20-76
SD5	93	—	48	60-150	92	16-160

\* LE = Liquid extrusion; CP = critical pressure method; IM = image analysis.

\*\* Only maximum pore size is measured.



**Figure 9.** Pores in Nyco 4.5 Fabric Observed with the Image Analysis System.



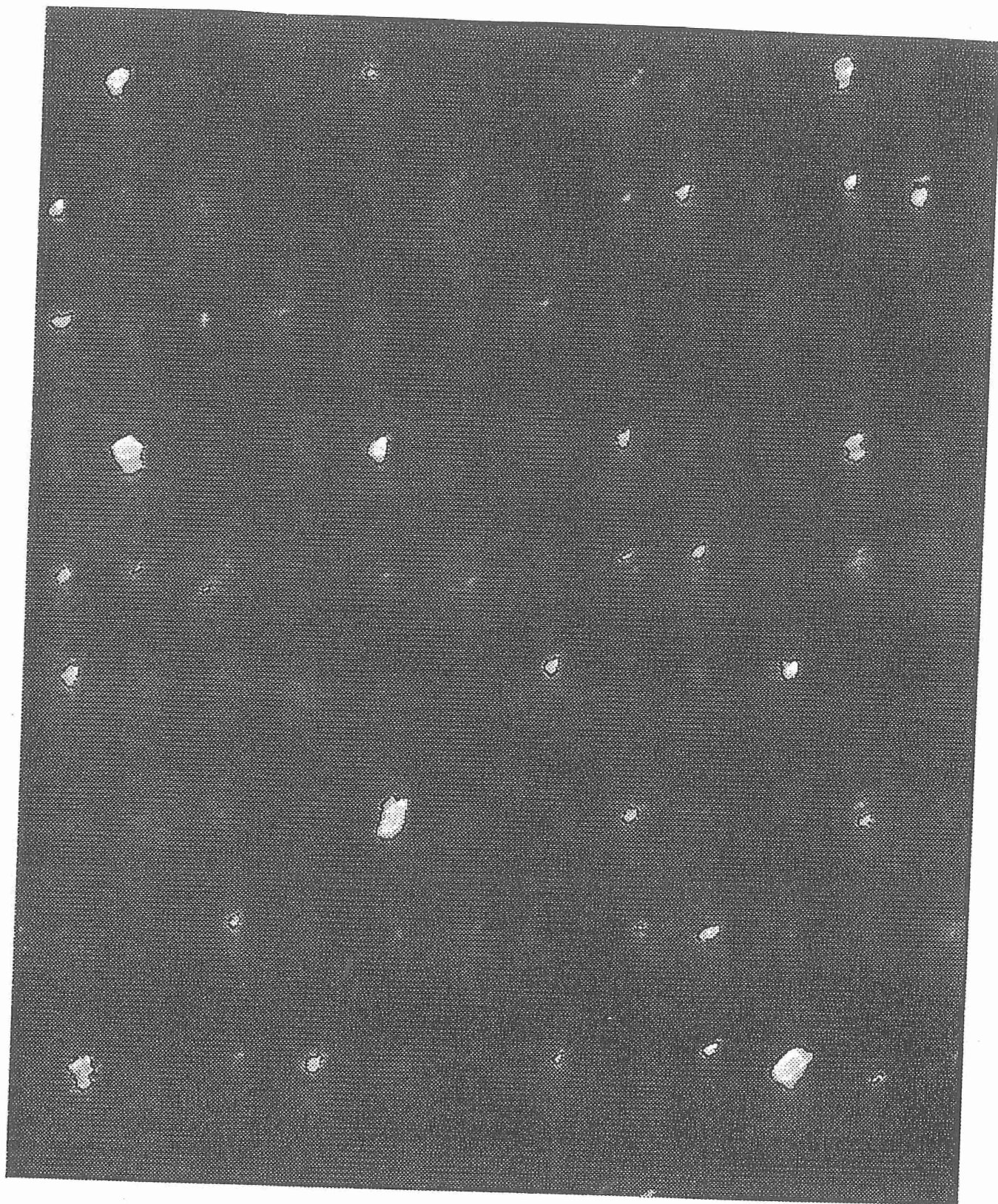


Figure 10. Pores in Nyco 7 Fabric Observed with the Image Analysis System.

### 3.3 Aerosol Penetration

The fabric penetration measurements focused on three general areas:

- (1) penetration of 2.21  $\mu\text{m}$  diameter particles through all nine test fabrics,
- (2) penetration of five different diameter particles (0.60  $\mu\text{m}$ –4.5  $\mu\text{m}$ ) through fabrics SD1–SD5, and (3) the effect of challenge time on particle penetration through fabrics SD1–SD5.

#### (a) Penetration of 2.21 $\mu\text{m}$ latex spheres

The first particle penetration measurements were made on the nine fabrics listed in Table 1 using fluorescent polystyrene spheres having a diameter of 2.21  $\mu\text{m}$ . A challenge flow rate of 0.1  $\text{ft}^3/\text{min}$  was used for the study [face velocity of 3.6  $\text{ft}/\text{min}$  (1.8  $\text{cm}/\text{s}$ )]. Three replicate measurements were made on each of the nine fabrics. The results are reported in Table 1.

Tyvek has the lowest penetration (0.2%) and the only measurable pressure drop (5"  $\text{H}_2\text{O}$ ) of all the test fabrics. The small size of the pores in Tyvek (see Table 2) is undoubtedly responsible for the low penetration and measurable pressure drop. Among the eight woven fabrics, SD2 had the lowest penetration (29.0%) and Nyco 4.5 fabric, which has the largest pore size of the nine fabrics, had the highest penetration (62.6%).

Although the mean pore size and size range of fabric SD1 is considerably greater than those of SD2, the aerosol penetrations through these two fabrics is similar (32.6% and 29.0%, respectively) indicating that pore size is not a parameter which singularly controls fabric penetration. Also, fabrics SD2 and SD3 have similar pore sizes (49.3 and 47.0  $\mu\text{m}$  diameter, respectively, measured by image analysis) and ranges of pore sizes, yet SD3 has much higher penetration (37.2%) than SD2 (29.0%). At the same time, however, the number of pores per unit area of SD3 is less than that of SD2 (180 and 247, respectively\*) which would suggest that the penetration through SD3 should be less than that through SD2. Thus there must be fabric material properties other than the number and size of pores which affect particle penetration. One such factor might be the areal density of the fabrics. Accordingly, aerosol penetration is plotted as a function of areal density in Figure 11. These data suggest that particle penetration through a fabric is related to the fabric areal density, penetration decreasing with increasing fabric areal density. Fabric structural design might also play a role, since penetration is greater through twill fabrics than through plain weave fabrics having the same weight per unit area.

#### (b) Penetration as a function of aerosol diameter

The penetration of five different size diameter aerosols through fabrics SD1–SD5 was measured. The diameters of the aerosols evaluated were 0.60  $\mu\text{m}$ , 1.01  $\mu\text{m}$ , 2.21  $\mu\text{m}$ , 2.87  $\mu\text{m}$  and 4.50  $\mu\text{m}$ . The challenge flow rate again was 0.1  $\text{ft}^3/\text{min}$ . Results of these measurements are summarized in Table 3 and are also plotted in Figure 12, which indicates that the penetration vs. particle diameter curves are similar in shape for all five fabrics. Penetration first increases with increasing

\* data from July-August 1992 Monthly Progress Report

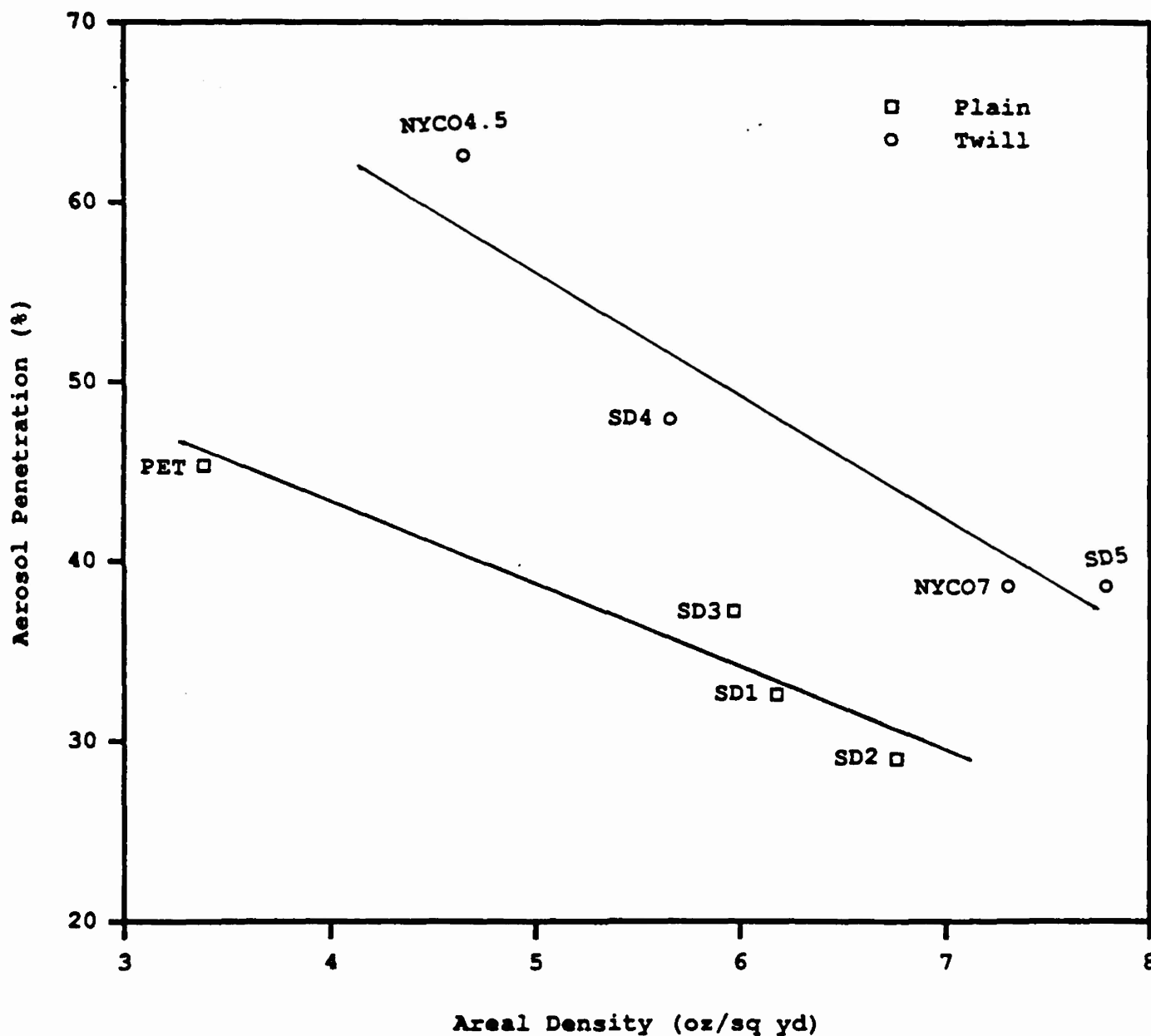
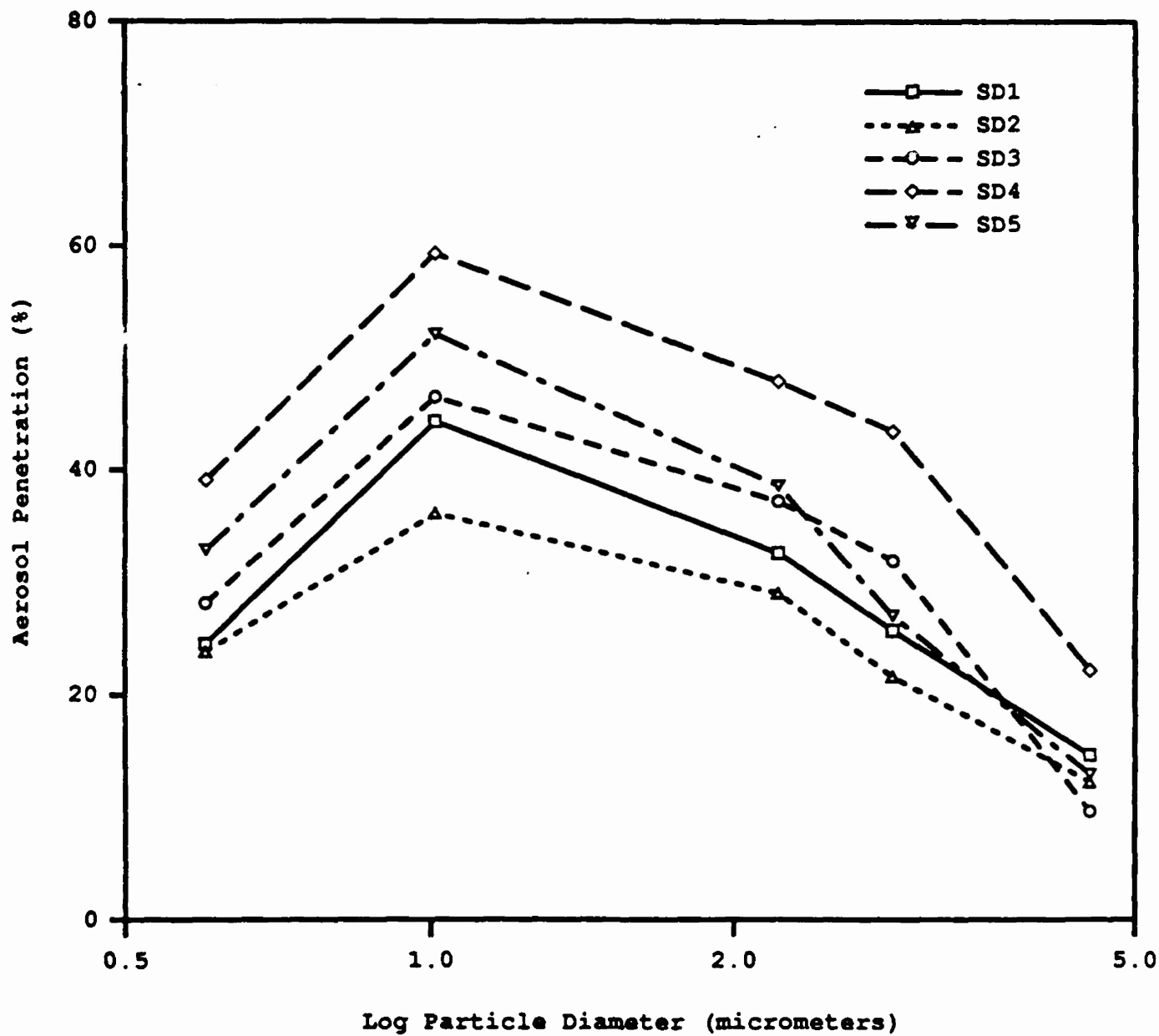


Figure 11. Effect of Fabric Areal Density and Structure on Penetration of 2.21  $\mu\text{m}$  Diameter Latex Spheres.



**Figure 12.** Penetration of Latex Spheres through Fabrics SD1-SD5 as a Function of Particle Diameter.



particle size, showing the influence of the diffusion mechanism at small diameters [4]. After reaching a maximum at approximately 1  $\mu\text{m}$  diameter, the penetration then decreases with increasing particle size, showing the increasingly important role of the inertial and impaction mechanisms [4]. The results obtained here confirm the well developed theories of aerosol mechanics and are similar to aerosol filtration studies by Fedele [5], Hinds [6], Lee [7], and VanOsdell [8].

**Table 3. Penetration of Polystyrene Sphere Aerosols through Fabrics**

Aerosol Diameter ( $\mu\text{m}$ )	Particle Penetration (%)				
	SD1	SD2	SD3	SD4	SD5
0.60	24.4	23.8	28.1	39.1	32.9
1.01	44.3	36.1	46.5	59.3	52.1
2.21	32.6	29.0	37.2	47.9	38.6
2.87	25.7	21.6	31.9	43.4	27.0
4.50	14.7	12.3	9.7	22.2	13.0

The rank order of the five fabrics in their resistance to aerosol penetration is consistent for the three smaller size aerosols (0.60, 1.01, and 2.21  $\mu\text{m}$  diameter); above this diameter there is one reversal in order at 2.87  $\mu\text{m}$  diameter and one change in the order with the 4.50  $\mu\text{m}$  particles in which fabric SD3 decreases in rank order from third highest to the lowest penetration. Fabric SD4 had the highest penetration for all particle diameters. Fabric SD2 had the lowest penetration of all the fabrics reported except for the highest diameter aerosol.

(c) Effect of challenge time on penetration

Figure 13 shows the dependence of particle penetration on challenge time for fabrics SD1 through SD5 for an aerosol of diameter 2.21  $\mu\text{m}$ . The flow rate is again 0.1  $\text{ft}^3/\text{min}$ , and the aerosol concentration is about 25,000/ $\text{ft}^3$  (0.88/ $\text{cm}^3$ ). Particle penetration of each fabric remained fairly constant over the test period of 40 minutes. Changes were less than  $\pm 3\%$  and tended to increase slightly with time. The pressure drop across the fabrics remained 0.0 inches throughout. These results are different from that reported by the Army Natick Laboratory in which a marked decrease in particle penetration occurred with increasing challenge time (on the order of 60% drops after 10 minutes) [9]. The drop in penetration was accompanied by an increase in pressure drop. This difference in behavior can probably be attributed to the much higher aerosol concentrations used at the Natick RD & E Center of 500/ $\text{cm}^3$ , about 570 times greater than that used here (0.88/ $\text{cm}^3$ ). Also, their face velocity (3.76  $\text{cm}/\text{sec}$ ) was 2.1 times greater than ours (1.8  $\text{cm}/\text{sec}$ ). Thus, the particle flux (particles/ $\text{cm}^2/\text{sec}$ ) through the fabric at Natick would be  $570 \times 2.1 = \sim 1200$  times greater than the flux used here. Since the flux (per unit sample area) is equal to the surface velocity times the particle concentration, with this higher

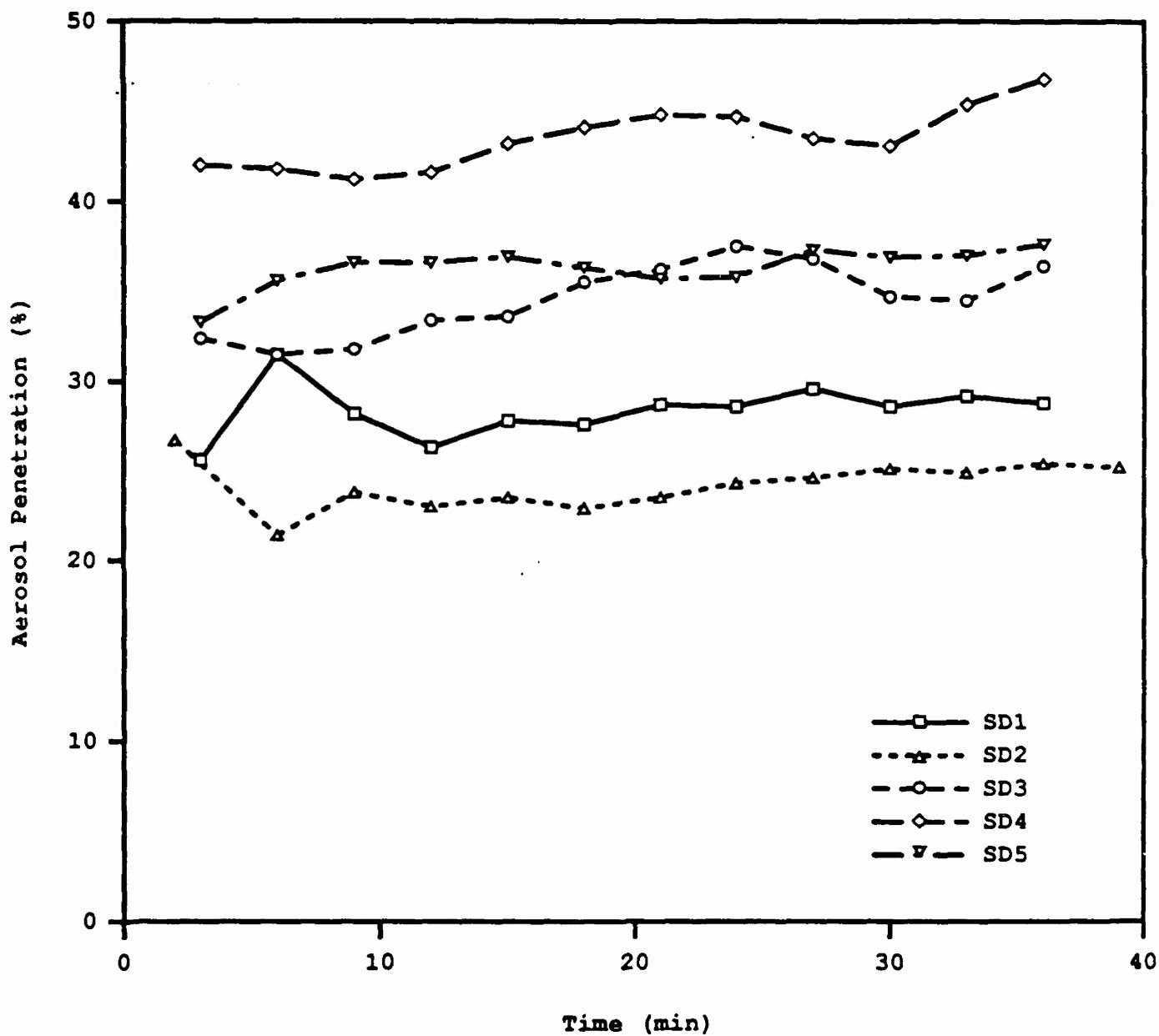


Figure 13. Effect of Challenge Time on Particle Penetration of 2.21  $\mu\text{m}$  Diameter Latex Spheres through Fabrics SD1-SD5.

flux of larger aerosol particles (2.7 vs 2.2  $\mu\text{m}$  diameter), it is certainly conceivable that clogging of pores might occur in the Natick studies well before any signs of clogging would appear in our studies, especially since the Natick challenge aerosol contains particles with diameters up to 4.5  $\mu\text{m}$ . In any event, particle penetration remains constant with zero pressure drop during the 40-minute challenge tests at NCSU.

#### 4.0 CONCLUSIONS

The major results from this study are as follows:

4.1 Pore size distributions measured independently by liquid extrusion, image analysis, and critical breakthrough pressure methods for fabrics yielded fairly consistent results with only a few differences in the rank order of the fabrics. Since the principles of the three methods are quite different, it is difficult to determine at this time which method might be preferred or whether each contributes useful independent information. Therefore, additional work and analyses will be made before one of these techniques will be recommended as a standard.

4.2 Many fabric structural factors such as weave pattern, fabric count, and yarn size affect particle penetration. As a first approximation, however, the particle penetration through a fabric might be related to the fabric density and weave type. The penetration was found to increase with decreasing fabric weight per unit areal density.

4.3 Results of aerosol penetration measurements as a function of particle diameter through selected fabrics indicate that the penetration vs. particle diameter curves are similar in shape for various fabrics. Penetration first increases with increasing particle size, showing the influence of the diffusion mechanism at small diameters. After reaching a maximum value, the penetration then decreases with increasing particle size, showing the increasingly important role of the inertial and impaction mechanisms.

#### 5.0 FUTURE WORK

5.1 Relationships between particle penetration and fabric design will be examined in more detail to provide additional understanding of how fabric structure can be improved to better resist penetration and still maintain comfort. These studies will include a more detailed examination of the influence of pore size and shape, their number, and their three-dimensional characteristics such as tortuosity.

5.2 Effects of surface velocity and particle concentration will be considered in more detail.

5.3 Discussions will continue with the Natick RD & E Center to keep the project oriented towards their needs.

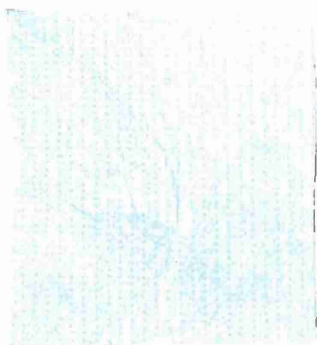
This document reports research undertaken at the U.S. Army Natick Research, Development and Engineering Center and has been assigned No. NATICK/TR-93/040 in the series of reports approved for publication.

## 6.0 REFERENCES

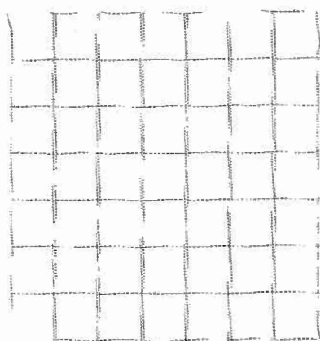
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**APPENDIX**  
**FABRIC SAMPLES EXAMINED IN THIS REPORT**

Appendix. Fabric samples examined in this report.



Tyvek



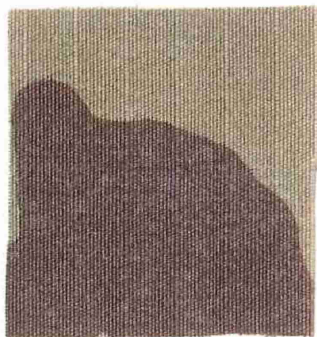
PET woven



Nyc 4.5



Nyc 7



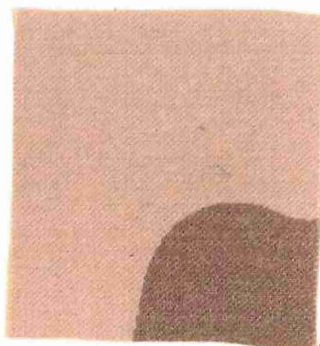
SD1



SD2



SD3



SD4



SD5